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[54] **CHEMICAL VAPOR DEPOSITION COATING OF FIBERS USING MICROWAVE APPLICATION**

[56]

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[51] Int. Cl.⁷ C23C 16/00; H05B 6/70

[52] U.S. Cl. 118/723 MW; 118/724; 118/722; 219/690

[58] Field of Search 118/723 MW, 118/715, 724, 722; 219/690, 691, 692, 693, 764, 695, 696, 697; 156/345

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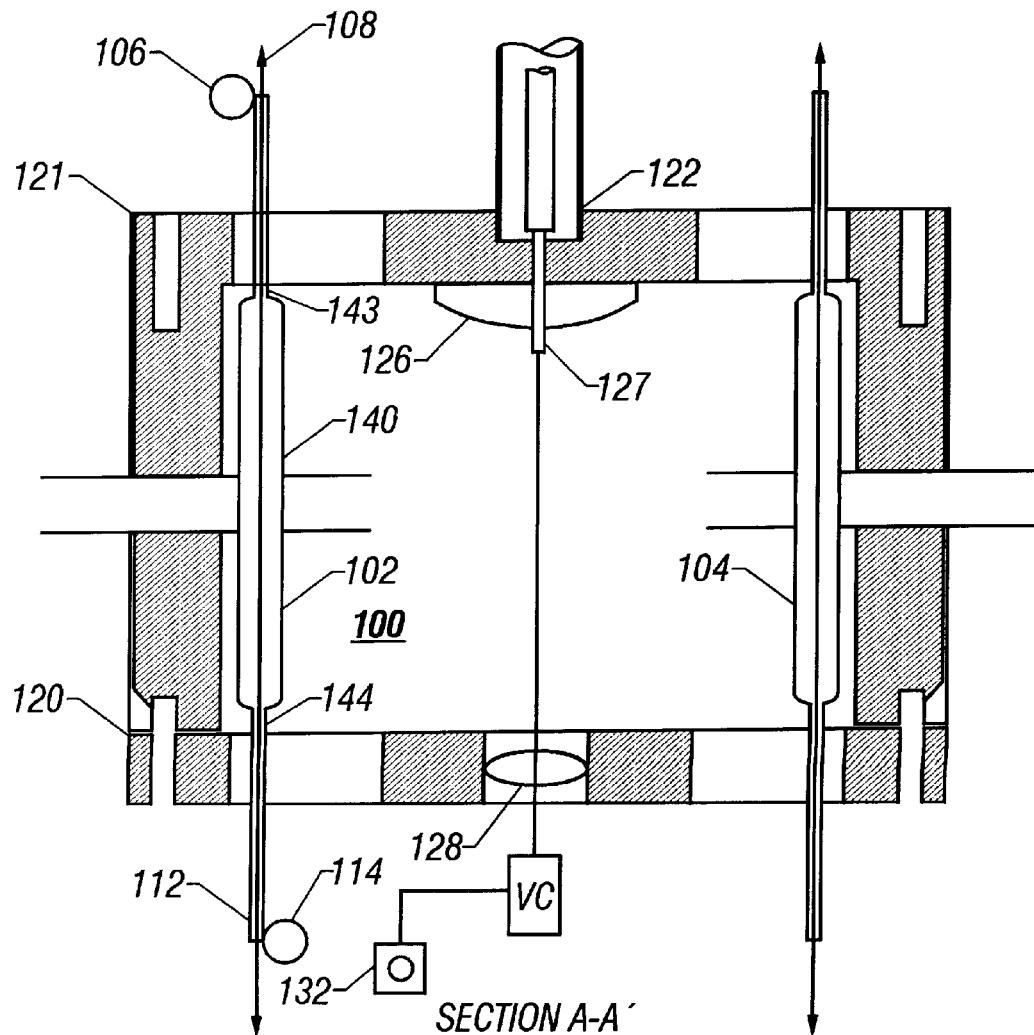
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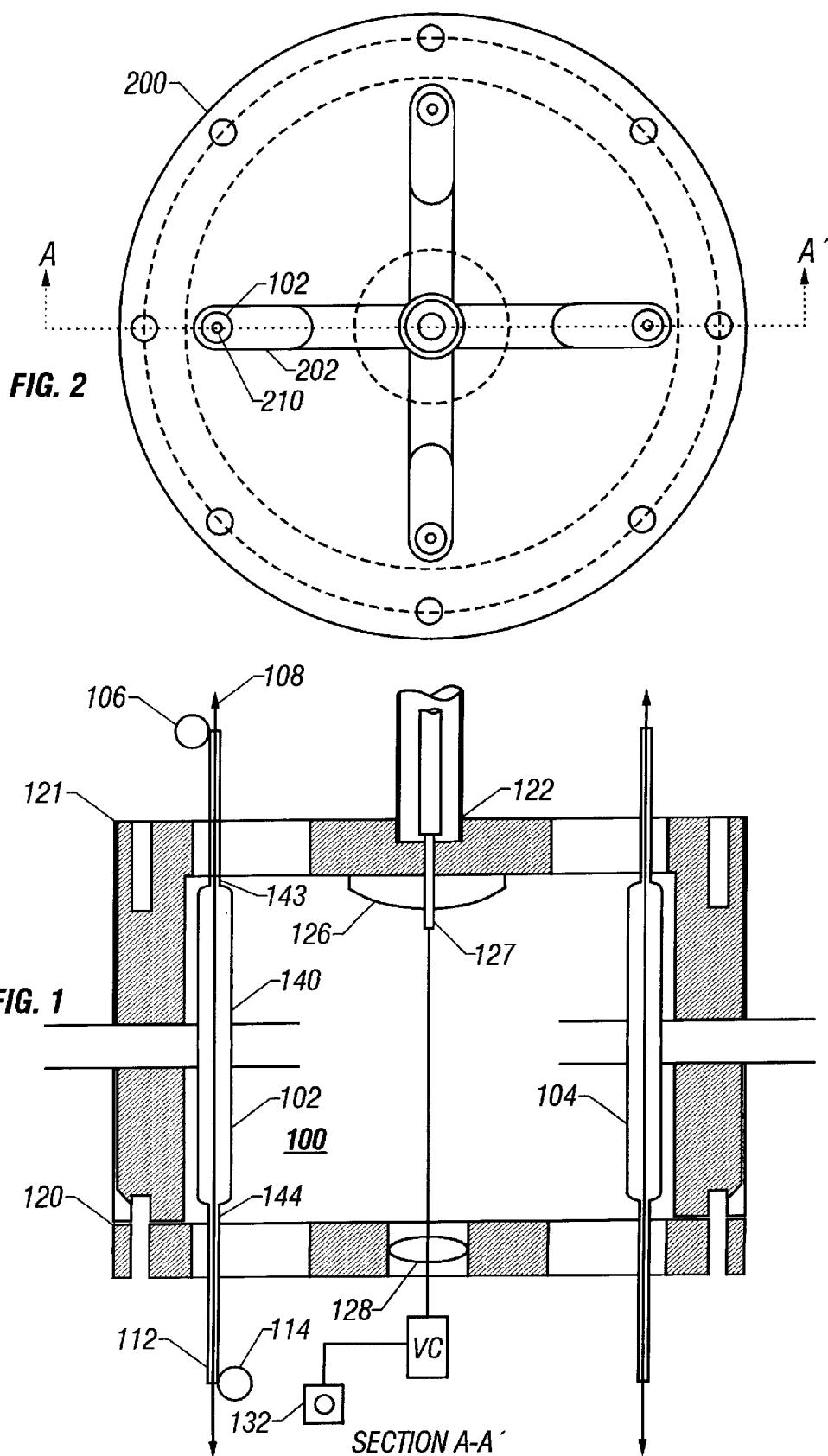
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ABSTRACT

Chemical vapor deposition coating is carried out in a cylindrical cavity. The fibers are heated by a microwave source that is uses a TM_{0N0} mode, where O is an integer, and produces a field that depends substantially only on radius. The fibers are observed to determine their heating, and their position can be adjusted. Once the fibers are uniformly heated, a CVD reagent is added to process the fibers.

13 Claims, 1 Drawing Sheet





1

**CHEMICAL VAPOR DEPOSITION COATING
OF FIBERS USING MICROWAVE
APPLICATION**

**CROSS REFERENCE TO RELATED
APPLICATIONS**

This application claims the benefit of the U.S. Provisional Application Ser. No. 60/086,833, filed on May 26, 1998.

**STATEMENT AS TO FEDERALLY SPONSORED
RESEARCH**

The invention described herein was made in the performance of work under a NASA contract, and is subject to the provisions of Public Law 96-517 (35 U.S.C. 202) in which the Contractor has elected to retain title.

BACKGROUND

Many different applications for coating of fibers are known. One way to coat a fiber is by chemical vapor deposition or "CVD".

Conventional coating of a fiber starts with a portion of the fiber called a core being heated by a direct electrical current through the fiber. The hot core is exposed to reagent vapor, to be coated by CVD. The core can be simultaneously spooled through a chamber containing reagent gases. The current is introduced into the chamber via mercury electrodes that also serve as gas seals for the ends of the reaction chamber. Each chamber can typically accommodate only a single fiber at a time.

Such a system has drawbacks. The toxic character of the mercury that is used in the electrodes becomes an environmental hazard. Moreover, many systems allow only a single fiber to be processed at a time. This can be expensive in terms of production costs. These systems also require that the fiber be at least a moderately good electrical conductor in order to achieve adequate heating for the CVD.

European Patent No. 0552951A1 describes coating fibers using CVD in a microwave heating of a waveguide applicator. This system operates without mercury and extends the range of fiber materials. However, this system is limited. For example, this system does not teach how to coat more than a single fiber at a time.

SUMMARY

The present system addresses this problem by coating fibers using CVD in a special, adjustable microwave cavity. The disclosed system uses CVD in a way that allows simultaneous processing of multiple fibers. This is done in a cylindrical resonant microwave cavity that is excited in a transverse magnetic (TM) mode, eg one which is essentially TM₀₁₀, where n is a positive integer. The system can avoid using toxic metals such as mercury. Moreover, the cavity applicator includes multiple reaction chambers, one reaction chamber for each fiber. Each of the reaction chambers gets about the same amount of microwave energy along almost its entire length.

In preferred embodiments, the chambers are defined by a tube that is made of low-loss material such as quartz. A reagent gas flows therethrough to form the CVD coating when the fiber temperature is above some threshold value. Known techniques based on flowing gas are used to prevent air contamination in the reaction chamber. This also permits the fiber to move continuously through the chamber.

Fiber feed and take up can be provided by conventional spool and pulley arrangements or similar arrangements can

2

be carried out where one or both pulleys are replaced by guide tubes as described herein. Further details are described in the following.

BRIEF DESCRIPTION OF THE DRAWINGS

These and other aspects of the invention will now be described with reference to the attached drawings, in which:

FIG. 1 shows a diagram of the chamber system; and

FIG. 2 shows a cross section of the chamber along the line 2—2.

**DESCRIPTION OF THE PREFERRED
EMBODIMENT**

15 The preferred embodiment is shown in FIG. 1 with a cross section along the line 2—2 being shown in FIG. 2. The system of FIG. 1 enables economically and efficiently heating and coating fibers using chemical vapor deposition in a resonant microwave cavity applicator. This applicator is excited in a mode that is essentially TM₀₁₀. The fibers are positioned to cause only small deviations from the empty cavity normal mode electromagnetic field distribution. At the same time, these fibers absorb a large fraction of the power that is transmitted into the cavity.

20 As explained herein, this positioning becomes critical for fibers that have highly lossy cores, such as carbon, or other materials.

25 The TM₀₁₀ mode is a resonant mode that provides symmetry. The electric field distribution for a TM₀₁₀ mode varies substantially only in the radial direction. This feature promotes uniformity of processing along the length of the fiber and also simplifies the selection of optimum locations for uniformity. Each of the fibers is preferably located near a radial electric field node. In this embodiment, the electric field intensity is maximum on the axis and substantially zero at the cavity wall. The electrical field vector is parallel to the axis throughout the cavity for each TM₀₁₀ mode.

30 Whenever N is greater than 1, one or more electric field nodal cylindrical surfaces will be located away from the curved walls of the cavity. This enables using additional multiple nodes to simultaneously coat multiple fibers.

35 An embodiment is shown in FIG. 1. This embodiment is used to preferentially apply a coating, preferably a silicon carbide coating, to any desired number of fibers simultaneously. Each of the fibers is at a symmetrical radial location. This system uses a resonant microwave cavity 100 which operates in the TM₀₁₀ mode. Each fiber is located within a housing portion 102, 104. Each housing portion is supplied with gas reagent which allows CVD to be carried out when reaching the right temperature. Each housing portion 102 is attached to a pulley feed spool 106, which feeds the fiber 108 through the processing apparatus. The opposite end of the device is connected to aspirator 112 to suck out the reagent, and to a take up spool 114. The preferred fiber has cores formed of carbon with diameters of approximately 30 μm . The processed fibers have outer diameters of about 130 μm . The cavity is excited by a traveling wave tube ("TWT") 120 connected to a "N" type connector 122 which excites the TM₀₁₀ mode in the cavity at a frequency of approximately 2.45 gigahertz with about 300 watts of power. The cavity has a length of about 7½ centimeters from the top portion 121 to the bottom portion 122.

40 50 55 60 65 70 75 80 85 90 95 100 105 110 115 120 125 130 135 140 145 150 155 160 165 170 175 180 185 190 195 200 205 210 215 220 225 230 235 240 245 250 255 260 265 270 275 280 285 290 295 300 305 310 315 320 325 330 335 340 345 350 355 360 365 370 375 380 385 390 395 400 405 410 415 420 425 430 435 440 445 450 455 460 465 470 475 480 485 490 495 500 505 510 515 520 525 530 535 540 545 550 555 560 565 570 575 580 585 590 595 600 605 610 615 620 625 630 635 640 645 650 655 660 665 670 675 680 685 690 695 700 705 710 715 720 725 730 735 740 745 750 755 760 765 770 775 780 785 790 795 800 805 810 815 820 825 830 835 840 845 850 855 860 865 870 875 880 885 890 895 900 905 910 915 920 925 930 935 940 945 950 955 960 965 970 975 980 985 990 995 1000 1005 1010 1015 1020 1025 1030 1035 1040 1045 1050 1055 1060 1065 1070 1075 1080 1085 1090 1095 1100 1105 1110 1115 1120 1125 1130 1135 1140 1145 1150 1155 1160 1165 1170 1175 1180 1185 1190 1195 1200 1205 1210 1215 1220 1225 1230 1235 1240 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3

antenna is penetration into the cavity can be adjusted to enable better impedance matching to the cavity. Suitable adjustment allows the reflected power to be low as a few percent.

A mirror 126 provided in the cavity enables a user monitoring the operation to observe the light emitted from the heated fibers along most of their length. The mirror can be a thin curved mirror as shown. Light reflected from the mirror passes through a re-imaging lens 128 located at the other end of the cavity. This image can be either directly watched or captured by a video camera 130 as shown, and then displayed on a monitor 132.

This optical system enables optimization of fiber positions and also allows continuous monitoring of the processing chamber.

A cross section of the device along the line 2—2 is shown in FIG. 2. The processing chamber is substantially cylindrical, having curved cavity wall 200. The fibers are located in slots 202 which are at substantially equal angular intervals, but at the same radial distance. The centers of these slots are preferably located about 3 mm from the wall. The TM010 mode that is used herein has an electric field with a node at the wall. Therefore, the fibers are located about 3 mm from the node.

Each of the slots 202 include a transparent quartz tube 102 that is supplied with reagent gas. The tube wall 210 is approximately 1 mm thick. The wall also has a varied diameter portion, including enlarged portion 140 which is approximately 6 mm in magnitude over most of the area inside the cavity. Portions 142, 144 are narrowed towards each end. The radial slots 202 enable adjustment of the radial position of the fiber in the cavity. This can optimize the location of the fiber and tube surrounding the fiber. In this way, the materials can be located in an optimum location for the desired heating effect.

According to a preferred mode, the optimized position produces a Q value which is high enough to maintain the integrity of the desired mode, while providing sufficient heat to the fibers as desired.

In operation, microwave energy is applied to the fibers in order to bring them to a specified temperature. That specified temperature can be visually determined—i.e., it is approximately when the fibers emit a bright yellow light for the materials described above. Then, the positions of the fibers are radially adjusted, as described above, to cause them to emit with substantially equal brightnesses along their lengths. The fibers near the end plates are in narrower chambers which provide less insulation of the fibers. Hence, these are cooler near those end plates.

The optical system described in summary above includes the curved glass mirror 126, the lens 128, video camera 130 and video monitor 132. This enables viewing the entire image of all of the fibers substantially simultaneously in order to determine the uniformity of the glowing fibers. Fine adjustments within the slots enable positioning the tubes for more even heating. In fact, the inventors found that even changing the position by as little as 1 mm can produce significant effects on the spatial distribution, color and brightness of the heated portions of the fibers. The inventors also found that it was easier to adjust the fibers when they are closer to the walls, and it is harder to adjust the positions of the fibers when they are farther from the walls.

In addition, radial positions of the fibers that are near the cavity axis has relatively low Q values. This can produce a poor impedance match between the cavity, antenna and coaxial transmission line. The input power to the cavity was

4

therefore limited, and the fibers were not easily heated to high-enough temperatures to allow CVD. Therefore this system prefers the positioning discussed above.

An alternative embodiment excites the cavity in a TM020 mode. In this TM020 mode, an electric field node is produced at a position that is about half the distance between the axis and the cavity wall. An important advantage of this embodiment is that larger diameter tubes can be used for the reaction chambers. This allows more reagent to be used, and also allows better insulation of the fibers. These larger diameter tubes can be positioned close to the cylindrical nodal surfaces. In this case, the Q of the cavity can be sufficiently high for good impedance matching even when many more fibers are in the cavity.

Use of larger tubes also reduces the rate at which reactant material is deposited on the tube wall. This permits longer processing intervals between cleaning operations.

This configuration also enables the walls of the reaction chamber to be further from the cavity walls. This increased separation reduces the additional but uneven heating of reaction chamber walls by reflected thermal radiation and by perturbations of the electric field at the reaction chamber walls closest to the cavity walls. Therefore, the rate of accumulation of reactant materials on the quartz walls is substantially lowered because of, *inter alia*, the lower wall temperatures of the reaction chambers.

Hence, it is preferred that even larger cavities could be used in the future to further reduce this.

An important feature of the techniques described above is that simultaneous processing of multiple fibers becomes possible. This can reduce the manpower to form the fibers, since multiple fibers can be coated in one operation. In addition, however, operating costs are reduced since approximately the same amount of power is used to coat multiple fibers, while in previous art that power was used to coat only a single fiber. In part, this higher efficiency is obtained by this system which better concentrates the microwave energy where it is needed, rather than on the cavity walls. Additional advantages are obtained by avoiding wasting power in a dummy load such as in a microwave waveguide applicator.

More efficient use of power is obtained by obtaining a good impedance match between the partially filled cavity and the antenna and transmission line that feeds it. The electric dipole rod antenna which is described herein extends into the cavity. This enables and facilitates adjustment provided that the fibers are properly positioned. With the preferred fibers which use lossy cores such as carbon, fiber positions are near an electric field node for the cylindrical TM010 mode. The optimum radius is further from the node for less lossy fibers. Better uniformity is obtained when the fibers are distributed at equal angular intervals around the circle as preferred.

The slots which are used allow optimization by adjustment of the radial position of the fiber. Additional optimization to achieve uniformity in processing can be obtained by the flow meter and control valves and fluid lines as shown.

As described above, the proper selection of the radius of the tubes also provides certain advantages. Larger radii provide the unexpected advantage that reactant material is slower to accumulate on the surface of the tube. This enables less frequent cleaning. A preferred optimum value is approximately 2 to 3 mm. However, even larger tubes can be used where their dimension are compatible with the size of the device being considered.

5

Although only a few embodiments have been described in detail above, other modifications are within the following claims. For example, other materials could be used for the cores, and this system is not limited to lossy cores. Also, other microwave frequencies and other sizes could be used.

What is claimed is:

1. A fiber chemical vapor deposition apparatus comprising:

a cavity;

a microwave source producing a microwave energy in said cavity, said microwave energy forming a transverse magnetic resonant mode within said cavity; and a plurality of fiber holding areas, within said cavity, each adapted for holding a fiber, each of said fiber holding areas having an adjustment, which allows a position of said fiber to be changed to allow said fibers to be heated substantially uniformly by said microwave energy; and an element, in said cavity, which provides reagent for chemical vapor deposition to said fibers in a presence of heating of said fibers from said microwave.

2. An apparatus as in claim 1 wherein said cavity is cylindrical.

3. An apparatus as in claim 2 wherein said adjustment comprises a slot within the cylindrical cavity which adjusts resonance to optimize microwave energy transmission to the fiber.

4. An apparatus as in claim 2 wherein said transverse magnetic mode is one in which energy of the radiation is substantially dependent only on radius, wherein said adjustment comprises a slot which enables adjusting a radius at which a fiber is located.

5. An apparatus as in claim 2 wherein said microwave source includes an antenna, and said antenna is tunable to match to said cavity.

6

6. An apparatus as in claim 1 further comprising a viewing element which enables viewing the fibers.

7. An apparatus as in claim 6 wherein said viewing element comprises a window and a lens which enables viewing the position of the fibers.

8. An apparatus as in claim 6 wherein said viewing element comprises a video camera and a display monitor.

9. An apparatus as in claim 1 wherein said microwave source includes an antenna, and wherein said antenna is tunable.

10. An fiber chemical vapor deposition apparatus, comprising:

a cylindrical cavity;

a microwave source, having an antenna which has an adjustable feature to enable matching to said cavity to produce resonant radiation within said cavity;

a plurality of tubes, located in said cavity, each said tube adapted for holding a fiber to be coated, each of said tubes having an adjustment, which allows a position of said fiber to be changed to allow said fibers to be heated substantially uniformly by said radiation; and

a reagent producing device, producing reagent into said tubes, said reagent of a type which causes a chemical vapor deposition in the presence of heating.

11. An apparatus as in claim 10 wherein said microwave source produces a transverse magnetic field which is dependent substantially only on radius within the cylindrical cavity.

12. An apparatus as in claim 10 further comprising a viewing element allowing observing the tubes.

13. An apparatus as in claim 10 further comprising a microwave tuning device which tunes an amount of microwave applied to said tubes.

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